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We Claim:

1. A process for the preparation of herbicidally effective formulations of
5 clomazone having a volatility less than fifty percent of the volatility of an emusifiable
concentrate of clomazone containing four pounds of clomazone per gallon of formulation
which comprises microencapsulating the clomazone by interfacial polymerization by the
steps of:
- 10 a) providing an aqueous phase containing 0.3 to 3.0 wt. % of one or more
emulsifiers; [optionally 0.02 to 0.20 wt. % of a xanthan gum viscosity
modifier/stabilizer, and 0.1 to 1.0 wt. % of an antifoam agent;]
b) providing a water immiscible phase consisting of clomazone, polymethylene
polyphenyl isocyanate (PMPPi), and a hydrocarbon solvent; the weight ratio of
15 clomazone to PMPPi being in the range of 1:1 to 6:1;
c) emulsifying the water immiscible phase in the aqueous phase, forming a
dispersion of water immiscible droplets throughout the aqueous phase;
d) agitating the dispersion while adding thereto an aqueous solution of 15 to 100
weight percent of at least one polyfunctional amine selected from
ethylenediamine (EDA), diethyltriamine (DETA), triethylenetetramine (TETA),
20 and 1,6-hexanediamine (HDA), with the proviso that (EDA) is used only in a
mixture, the weight ratio of polyfunctional amine to PMPPi being in the range of
0.1:1 to 1:1, thus forming microcapsules having a polyurea shell wall around the
water immiscible droplets; and
e) curing the microcapsules by continuing the agitation while heating the dispersion
25 at a temperature in the range of 35° to 60° C. for a period of 3 to 10 hours to
produce a formulation in which the average size of the microcapsules is in the
range of 5 to 50 microns [;
f) optionally adjusting the pH to between 6.5 and 9.0] .

- 30 2. A process according to claim 1 in which the emulsifier is a polyvinyl alcohol;
the antifoam agent is a polydimethyl siloxane; the ratio of clomazone to PMPPi is 4.5:1

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to 4.7:1; the polyamine is a mixture of TETA and HDA in which the ratio of TETA to HDA is 3:1 to 1:3; the microcapsules are cured at 45° to 50° C. for 4 to 5 hours and have an average size of 5 to 30 microns.

- 5 3. A process of claim 2 in which there is added to the formulation after completion of the curing step one or more stabilizers selected from 0.05 to 0.30 wt. % xanthan gum, 0.75 to 6.5 wt. % propylene glycol, 0.5 to 6.0 wt. % one or more surfactants, and 0.25 to 0.50 wt. % smectite clay, to adjust the viscosity to 1700 to 3800 cps and the suspensibility to greater than 70%, each weight percent relative to the weight
10 of the formulation after addition of the stabilizers.

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15 4. A process according to claim 1 in which the emulsifier is a polyvinyl alcohol; the antifoam agent is a polydimethyl siloxane; the ratio of clomazone to PMPPI is 4.5:1 to 4.7:1; the polyamine is a mixture of DETA and HDA in which the ratio of DETA to HDA is 3:1 to 1:3; the microcapsules are cured at 45° to 50° C. for 4 to 5 hours and have an average size of 5 to 30 microns.

- 20 5. A process of claim 3 in which the amounts of stabilizers added are 0.05 to 0.25 xanthan gum and 1.0 to 6.0 propylene glycol.

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25 6. A process according to claim 1 in which the emulsifiers are a polyvinyl alcohol [and, optionally a sodium salt of sulfonated naphthalene condensate] ; the antifoam agent is a polydimethyl siloxane; the ratio of clomazone to PMPPI is 4.5:1 to 4.7:1; the polyamine is DETA, the microcapsules are cured at 45° to 50° C. for 4 to 5 hours and have an average size of 5 to 30 microns.

- 30 7. An herbicidal formulation prepared according to any one of claims 1 through 6.

8. A process for the preparation of herbicidally effective formulations of clomazone having a volatility less than fifty percent of the volatility of an emusifiable

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concentrate of clomazone containing four pounds of clomazone per gallon of formulation which comprises microencapsulating the clomazone by interfacial polymerization by the steps of:

- 5 a) providing an aqueous phase containing 0.5 to 3.0 wt. % of one or more emulsifiers; [optionally 0.05 to 0.20 wt. % of a xanthan gum viscosity modifier/stabilizer, and 0.3 to 1.0 wt. % of an antifoam agent;]
- b) providing a water immiscible phase consisting of clomazone, polymethylene polyphenyl isocyanate (PMPPI), and a hydrocarbon solvent; the weight ratio of clomazone to PMPPI being in the range of 1:1 to 6:1;
- 10 c) emulsifying the water immiscible phase in the aqueous phase, forming a dispersion of water immiscible droplets throughout the aqueous phase;
- d) agitating the dispersion while adding thereto at least one polyfunctional amine selected from diethyltri-amine (DETA), triethylene-tetramine (TETA) and 1,6-hexanediamine (HDA), the weight ratio of polyfunctional amine to PMPPI being
- 15 in the range of 0.1:1 to 1:1, thus forming microcapsules having a polyurea shell wall around the water immiscible droplets; and
- e) curing the microcapsules by continuing the agitation while heating the dispersion at a temperature in the range of 35° to 60° C. for a period of 3 to 10 hours [;
- f) optionally adjusting the pH to between 6.5 and 9.0].

20 9. An herbicidal [composition] formulation containing from 1 to 4 pounds of clomazone per gallon of formulation and having a volatility less than fifty percent of the volatility of an emusifiable concentrate of clomazone containing four pounds of clomazone per gallon of formulation, comprising:

- 25 a) an aqueous suspension of microcapsules made up of a polyurea shell surrounding a core of clomazone and a minor amount of a hydrocarbon solvent, the polyurea having been formed from the interfacial reaction of polymethylene polyphenyl isocyanate (PMPPI) with ethylenediamine (EDA), diethylenetriamine (DETA), triethylenetetramine (TETA), or 1,6-hexanediamine (HDA), or a mixture of the
- 30 polyfunctional amines, with the proviso that EDA is used only in a mixture;
- b) 0.2 to 1.00 wt. % polyvinyl alcohol;

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- c) 0.1 to 0.5 wt. % antifoam agent; and
d) ¹⁷¹optionally 0.07 to 0.30 wt. % xanthan gum viscosity modifier/stabilizer; and
e) ¹⁷³0.75 to 7.0 wt. % propylene glycol, the average size of the microcapsules being
in the range of 5 to 50 microns and having a suspensibility of greater than 70%, a
5 viscosity of 1700 to 3800 cps, and a 100 mesh wet screen analysis of greater than
99.95%.

10. A ¹⁷¹[composition] ¹⁷³formulation of claim 9 containing two pounds of clomazone
per gallon of formulation, in which the weight ratio of clomazone to PMPPI is 4.5:1 to
10 4.7:1 and the polyfunctional amines are TETA and HDA, with the weight ratio of TETA
to HDA 3:1 to 1:3.

11. A ¹⁷¹[composition] ¹⁷³formulation of claim 9 containing two pounds of clomazone
per gallon of formulation, in which the weight ratio of clomazone to PMPPI is 4.5:1 to
15 4.7:1 and the polyfunctional amines are TETA and DETA, with the weight ratio of
TETA to DETA 3:1 to 1:3.

12. A ¹⁷¹[composition] ¹⁷³formulation of claim 9 containing two pounds of clomazone
per gallon of formulation, in which the weight ratio of clomazone to PMPPI is 4.5:1 to
20 4.7:1 and the polyfunctional amines are DETA and HDA, with the weight ratio of DETA
to HDA 3:1 to 1:3.

13. A ¹⁷¹[composition] ¹⁷³formulation of claim 9 containing three pounds of
clomazone per gallon of formulation, in which the weight ratio of clomazone to PMPPI
25 is 4.5:1 to 4.7:1 and the polyfunctional amine is DETA.

14. A ¹⁷¹[composition] ¹⁷³formulation of claim 13 in which the pH is adjusted to
between 6.5 and 9.0.

30 ¹⁷¹I 15. The process for the preparation of clomazone formulations of claim 1 further
comprising:

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f) adjusting the pH of the cured microcapsule dispersion to between 6.5 and 9.0.

5 16. The process for the preparation of clomazone formulations of claim 1,
wherein the aqueous phase of step a) comprises 0.02 to 0.20 wt. % of a xanthan gum
viscosity modifier/stabilizer.

10 17. The process for the preparation of clomazone formulations of claim 16,
wherein the aqueous phase of step a) further comprises 0.1 to 1.0 wt. % of an antifoam
agent.

15 18. The process according to claim 6 in which the emulsifiers comprise a
polyvinyl alcohol and a sodium salt of sulfonated naphthalene condensate.

20 19. The process for the preparation of clomazone formulations of claim 8 further
comprising:

f) adjusting the pH of the cured microcapsule dispersion to between 6.5 and 9.0.

25 20. The process for the preparation of clomazone formulations of claim 8,
wherein the aqueous phase of step a) comprises 0.05 to 0.20 wt. % of a xanthan gum
viscosity modifier/stabilizer, and 0.3 to 1.0 wt. % of an antifoam agent.

30 21. The herbicidal formulation of claim 9, further comprising:
e) 0.07 to 0.30 wt. % xanthan gum viscosity modifier/stabilizer.

22. An herbicidal formulation comprising an aqueous suspension of microcapsules,
wherein said microcapsules comprise a polyurea shell and encapsulated material
comprised of an herbicidally effective amount of clomazone and a hydrocarbon
solvent,
wherein the formulation has a clomazone volatility less than the volatility of an
emusifiable concentrate of clomazone containing a corresponding concentration of
clomazone.

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23. The herbicidal formulation of claim 22, wherein the clomazone volatility is less than fifty percent that of the emulsifiable concentrate of clomazone.

24. The herbicidal formulation of claim 22, wherein the formulation has from 1 to 4 pounds of clomazone per gallon of formulation.

25. The herbicidal formulation of claim 24, wherein the average size of the microcapsules ranges from 5 to 50 microns.

26. The herbicidal formulation of claim 25, wherein the formulation has a suspensibility of greater than 70%.

27. The herbicidal formulation of claim 26, wherein the formulation has a viscosity of 1700 to 3800 cps.

28. The herbicidal formulation of claim 27, wherein the formulation a 100 mesh wet screen analysis of greater than 99.95%.

29. The herbicidal formulation of claim 22, wherein the average size of the microcapsules ranges from 5 to 30 microns.

30. The herbicidal formulation of claim 22, wherein the encapsulated material comprises 60 to 85 weight percent clomazone.

31. A process for the preparation of an herbicidally effective formulation of clomazone which comprises the steps of:

a) providing an aqueous phase;

b) providing a water immiscible phase comprising clomazone, polymethylene

polyphenyl isocyanate (PMPPI), and a hydrocarbon solvent, wherein the amount

of clomazone is sufficient to provide an herbicidally effective concentration in the product formulation;

c) emulsifying the water immiscible phase in the aqueous phase, forming a dispersion of water immiscible droplets throughout the aqueous phase;

5 d) agitating the dispersion while adding thereto an aqueous solution of at least one polyfunctional amine, thus forming microcapsules having a polyurea shell wall around the water immiscible droplets; and

e) curing the microcapsules,
thereby obtaining an aqueous suspension of microparticles containing an herbicidally
10 effective concentration of clomazone and having a volatility less than the volatility of an emusifiable concentrate of clomazone containing a corresponding concentration of clomazone.

15 32 The process of claim 31, wherein the clomazone volatility is less than fifty percent of the emusifiable concentrate of clomazone.

33. The process of claim 31, wherein the suspension has from 1 to 4 pounds of clomazone per gallon of formulation.

20 34. An herbicidal formulation comprising an aqueous suspension of microcapsules wherein said microcapsules comprise a polyurea shell and encapsulated material comprised of an herbicidally effective amount of clomazone and a hydrocarbon solvent, the polyurea having been formed from the interfacial reaction of polymethylene polyphenyl isocyanate (PMPPI) with one or more polyfunctional amines,

25 wherein the formulation has a clomazone volatility less than the volatility of an emusifiable concentrate of clomazone containing a corresponding concentration of clomazone.

30 35. The clomazone formulation of claim 34, wherein the polyfunctional amine is selected from the group consisting of ethylenediamine (EDA), diethyltriamine (DETA),

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triethylenetetramine (TETA), and 1,6-hexanediamine (HDA), with the proviso that (EDA) is used only in a mixture.

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36. The clomazone formulation of claim 34, wherein the formulation has from 1 to 4 pounds of clomazone per gallon of formulation.

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37. A microencapsulated clomazone comprising a shell, whereby said shell substantially reduces the volatility of clomazone.

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38. The microencapsulated clomazone of claim 37, wherein said shell is polyurea.

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39. The microencapsulated clomazone of claim 38, wherein said polyurea is formed by interfacial polymerization of PMPPI and a polyfunctional amine.

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40. The microencapsulated clomazone of claim 39, wherein the polyfunctional amine is selected from the group consisting of ethylenediamine (EDA), diethyltriamine (DETA), triethylenetetramine (TETA), and 1,6-hexanediamine (HDA), with the proviso that (EDA) is used only in a mixture.

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41. The clomazone formulation of claim 33, wherein the encapsulated material comprises 60 to 85 weight percent clomazone.

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42. The clomazone formulation of claim 33, wherein the volatility of the formulation is reduced compared to an emusifiable concentrate of clomazone containing a corresponding concentration of clomazone, such that when the formulation is applied to a target area, injury to plants in areas adjacent to the target area is significantly reduced.

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43. A process for the preparation of a herbicidally effective formulation of clomazone which comprises microencapsulating clomazone by the steps of:

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- a) providing an aqueous phase containing an effective amount of one or more emulsifiers, and an effective amount of an antifoam agent;
- b) providing a water immiscible phase consisting of clomazone, polymethylene polyphenyl isocyanate (PMPPI), and a hydrocarbon solvent;
- c) emulsifying the water immiscible phase in the aqueous phase, forming a dispersion of water immiscible droplets throughout the aqueous phase;
- d) agitating the dispersion while adding thereto an aqueous solution of at least one polyfunctional amine selected from ethylenediamine (EDA), diethyltriamine (DETA), triethylenetetramine (TETA), and 1,6-hexanediamine (HDA), with the proviso that (EDA) is used only in a mixture, thus forming microcapsules having a polyurea shell wall around the water immiscible droplets; and
- e) curing the microcapsules by continuing the agitation while heating the dispersion to produce a formulation in which the average size of the microcapsules is in the range of 5 to 50 microns,
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- thereby obtaining a aqueous suspension of microparticles containing from 1 to 4 pounds of clomazone per gallon of formulation and having a volatility less than fifty percent of the volatility of an emusifiable concentrate of clomazone containing a corresponding concentration of clomazone.
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~~44~~. The process of claim ⁴²~~43~~, wherein step d comprises:
- d') agitating the dispersion while adding thereto at least one polyfunctional amine selected from diethyltriamine (DETA), triethylene-tetramine (TETA) and 1,6-hexanediamine (HDA).

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~~45~~. The process of claim ⁴²~~43~~, wherein step d comprises:
- d') agitating the dispersion while adding thereto 1,6-hexanediamine (HDA).

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~~46~~. The process of claim ⁴²~~43~~, wherein the suspension has from 1 to 4 pounds of clomazone per gallon of formulation.

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47. The process for the preparation of a herbicidally effective formulation of
clomazone claim ⁴²43, wherein the volatility of the formulation obtained is reduced
compared to an emusifiable concentrate of clomazone containing a corresponding
concentration of clomazone, such that when the formulation is applied to a target area,
5 injury to plants in areas adjacent to the target area is significantly reduced.

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